

METHOD FOR PRODUCING TETRAPHENOXIDE-N-ANILOYX
CYCLO PHOSPHAZOTRIENE

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Translation of Sposob polucheniya tetrafenoksidi-n-aniloksi-
tsiklofosfazotriena, USSR Patent No. 320498, January 24, 1972
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16. Abstract A method for producing tetraphenoxide-n-aniloxycyclophosphazotriene, distinguished by the fact that tetraphenoxide dichlorocyclophosphazotriene is processed with an alkaline metal n-acetamidophenolate in an organic solvent, for example pyridine, during heating, after which the resulting compound is saponified in the presence of a mineral or organic acid, for example, hydrochloric, and the desired product separated by well-known techniques.			
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CYCLO PHOSPHAZOTRIENE

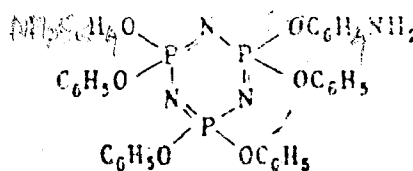
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Description

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The invention pertains to a method of producing nitrogen /1*
compounds, more precisely to a method of producing
tetraphenoxide-n-aniloxy cyclo phosphazotriene



There exists a method for obtaining tetraphenoxide-n-aniloxy
cyclo phosphazotriene through the reaction of hexachlorphos-
phazotriene with an alkaline metal n-nitrophenolate in an
organic solvent, after which the resulting product is reduced,
for example on Raney nickel in an autoclave at 80-90°C.

Literature offers no description of the production of
tetraphenoxide-n-aniloxy cyclo phosphazotriene or of the
compound itself, which are new. The substances produced can be
used in polymer chemistry to obtain heat-resistant
heteroaromatic polymers.

The method of producing tetraphenoxide-n-aniloxy cyclo
phosphazotriene involves processing tetraphenoxide dichloro-

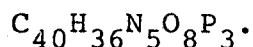
*Numbers in the margin indicate pagination of the foreign text.

cyclophosphazotriene with an alkaline metal n-acetamido-phenolate in an inert organic solvent, for example pyridine or xylene during heating, after which the resulting compound is saponified in the presence of a mineral or organic acid, for example hydrochloric, and the desired product is separated by well-known techniques.

The first stage takes place at 80-150°C; saponification, at 50-80°C.

Example 1. A mixture of 17.34 g (0.03 mole) tetraphenoxide dichlorocyclophosphazotriene, 9.1 g (0.06 mole) of n-oxyacetanilide, and 3.63 g (0.06 mole) of solid potassium hydroxide in 120 ml of pyridine is combined in a flask fitted with a reflux condenser, with the solvent boiling for 6 hr. After the potassium chloride is precipitated out, the cooled solution is boiled dry in a vacuum (20-30 mm Hg). The residue is a very viscous light-brown liquid which hardens into a resinous mass upon cooling. The product crystallized very slowly as it was left to stand. Crystallization can be greatly accelerated by adding a small amount of acetone or acetone-heptane mixture. In the latter case, tetraphenoxide-(n-acetaniloxide)-cyclophosphazotriene was separated as colorless prismatic crystals. Both the crystalline and resinous products were identified using ultimate analysis as tetraphenoxide-(n-acetaniloxide)-cyclophosphazotriene. The crystalline product's melting point was 160-162°C; yield, 85-92% (theoretical).

Found, %: C = 59.15; 59.05; H = 4.58, 4.46; N = 8.05, 8.40.



Calculated, %: C = 59.48; H = 4.49; N = 8.67.

The resinous tetraphenoxide-(n-acetaniloxide)-cyclo-

phosphazotriene was saponified and heated during mixing with 10 times its weight of 18% hydrochloric acid. Processing was done at 60°C for 3 hr. During saponification, the resinous product was converted into a solution.

At the end of saponification, the solution was cooled and processed in a 25% solution of potassium hydroxide. The light-brown resinous product separated during neutralization was rinsed in water at 40°C until a neutral reaction and dried in a vacuum drying cabinet. Ultimate analysis and infrared spectroscopy identified the product, which crystallizes when left to stand, as tetraphenoxide-n-aniloxide-cyclophosphazotriene with a melting point of 144-148°C; yield, 69-73% (theoretical), total yield 65%.

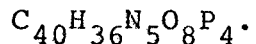
Found, %: C = 59.35, 59.18; H = 4.38, 4.25; N = 9.15, 9.48.

$C_{36}H_{illegible}N_5O_6P_{illegible}$

Calculated, %: C = 59.75; H = 4.46; N = 9.68.

Example 2. A mixture of 17.34 g (0.03 mole) tetraphenoxide dichlorocyclophosphazotriene, 9.1 g (0.06 mole) of n-oxyacetanilide, and 3.63 g (0.06 mole) of solid potassium hydroxide in 100 ml of xylol was prepared in a flask fitted with a reflux condenser with Dine-Stark trap; the solvent was then boiled for 8 hr. The water separated during the reaction was distilled and collected in the trap. When the reaction ended, a precipitate of potassium chloride was filtered from the cooled acid solution. It was boiled dry in a vacuum (20-30 mm Hg). After cooling, the residue was a resinous light-brown mass which crystallized slowly as it stood. Yield = 73-78%. According to results of ultimate analysis, the product was identified as tetraphenoxide-(n-acetaniloxide)-cyclophosphazotriene.

Found, %: C = 59.05, 59.42; H = 4.28, 4.35; N = 8.12, 8.01.

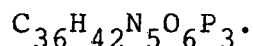


Calculated, %: C = 59.48; H = 4.49; N = 8.67.

The product was then processed as described in example 1.

After saponification, a resinous light-brown substance which crystallized slowly as it stood was produced. Melting point, 144-148°C; yield, 69-73%; total yield, 45%.

Found, %: C = 58.93, 59.12; H = 4.87, 4.55; N = 9.18, 9.33.



Calculated, %: C = 59.75, H = 4.46; N = 9.68.

SUBJECT OF THE INVENTION

1. A method for producing tetraphenoxide-n-aniloxo cyclo phosphazotriene, distinguished by the fact that tetraphenoxide dichlorocyclophosphazotriene is processed with an alkaline metal n-acetamidophenolate in an organic solvent, for example pyridine, during heating, after which the resulting compound is saponified in the presence of a mineral or organic acid, for example, hydrochloric, and the desired product separated by well-known techniques.

2. The method as per point 1, distinguished by the fact that the process takes place at 50-150°C.

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